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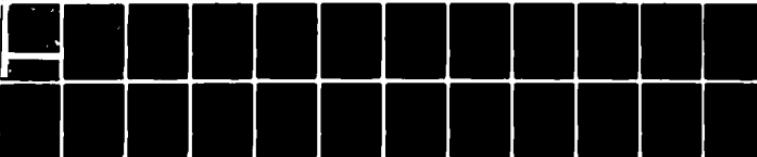
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JUL 81 J W MAYER, M NICOLET, J O MCCALDIN N00014-75-C-0912
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on

THE APPLICATION OF ION IMPLANTATION
TO COMPOUND FORMATION

N00014-75-C-0912 ✓

1 May 1975 to 30 April 1981

Principal Investigators

James W. Mayer

Marc-A. Nicolet

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July 1981

DISTRIBUTION STATEMENT A

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I. OVERVIEW

Contract N00014-75-C-0912 spanned a period of six years, from 1 May 1975 to 30 April 1981. There were three co-principal investigators in the first two years (J. W. Mayer, J. O. McCaldin and M-A. Nicolet), two in the subsequent three years (J. W. Mayer and M-A. Nicolet), and one (M-A. Nicolet) in the last year. The contract was monitored by D. K. Ferry and G. B. Wright. In the first two years, the contract focused on solid-phase epitaxy. In the final four years, the application of ion implantation to compound formation was the main topic of investigation. These many changes are indicative of the exciting times and the rapid progress that were spanned by this contract period. They also reflect the stresses and adjustments that went along with such a fast evolution.

During the whole period of the contract, other topics were pursued as well, e.g. the development of a radioactive tracer technique for the study of thin-film reactions; the development of a reliable current integration system for ion-beam systems; the development of Au standards for the calibration of BS systems; the development of very thin silicon single crystalline self-supported films. Several of these projects turned out to have a determining effect on the direction that the program took in subsequent years. These projects were also responsible for some of the major contributions that were ultimately made under this contract. In retrospect, it is clear that the flexibility that the principal investigators enjoyed in the pursuit of their work was one of the main reasons for the success of this contract. The supporting agency and the technical

monitors deserve high praise for their essential part in creating and maintaining the conditions that are necessary to assure such flexibility. Of these, continuity of support and common goals, priorities, and trust were paramount.

The listing of the publications sponsored under this contract, and of the many individuals involved in it, properly reflects the breadth of activity covered by the contract. In the following, we highlight those results that already have - or possibly still will - affect the development of future research in a significant way.

1. Solid-Phase Epitaxial Growth (SPEG).

Most of the facts now known about SPEG of Si and Ge through transport layers have been obtained under this contract. We predicted that SPEG of Si on silicon without a transport layer should be feasible - a prediction that has since been proven correct.

2. Radioactive ^{30}Si Tracer Technique.

The radioactive tracer technique developed under this contract is now systematically applied by others and furnishes among the most detailed quantitative information now available on the reactions of silicon with metal films or ambients during thermal annealing. These reactions are presently a topic of high interest in the semiconductor industry.

3. Redistribution of Impurities in Silicide Formation.

The possibility to use advantageously the redistribution of electrically active impurities that sometimes takes place during thermally induced silicide formation was formulated as a result of experiments performed under this contract. The idea has yet to be

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pursued systematically. It should and may well be quite important practically.

4. Ion Mixing.

Ion mixing - an outgrowth of our (proposed) studies of high-dose implantation - has attracted much interest and promises to become a complement to the presently accepted process of ion implantation.

5. Very Thin <Si> Films.

The development of very thin self-supporting Si single-crystal films of large (cm^2) area has opened up new ways of non-destructively analyzing with relative ease and with high depth resolution the atomic make-up of silicon single-crystal interfaces buried below 1000 \AA of a thin metal or dielectric layer. These very thin <Si> films are also useful for the analysis of thin-film reaction, ion mixing, and channeling phenomena that would be quite difficult to investigate otherwise.

6. 400 kV Ion Implantation System.

This contract financed the acquisition of an Accelerators, Inc. 400 kV ion implantation system that has been gradually improved and is now very probably the most versatile system of its kind in the nation's academic institutions. As a result of changes at Caltech and ONR, this system is presently without support. In view of the exciting new opportunities that can be seen for ion mixing in problems relating to surface ennoblement (e.g. electrochemistry, corrosion, tribology), thin films (amorphous materials, metastable phases) and electronic device technology (thin-film reactions, impurity-controlled phenomena, ohmic and rectifying contacts), this is a great pity. As

the title holder to this system, ONR should perhaps initiate an exchange of views with M-A. Nicolet to come to a consensus as to what should best be done. Progress in technologically important areas is at stake. Alternatives may present themselves that could be preferable to the present situation that evolved out of developments in which the existence and effectiveness of this powerful system was left out of considerations.

II. Summary of Scientific Results

1. Solid-Phase Epitaxy, Radioactive Tracer Technique, and Related Topics.

Backscattering spectrometry and scanning electron microscopy have been used to study the transport of Si from an amorphous Si layer ($\approx 1 \mu\text{m}$) through a Pd-silicide layer ($\approx 0.2 \mu\text{m}$) onto <100> oriented Si⁽⁹⁾. For a given annealing temperature, two distinct stages of this process have been observed. The initial "transient" stage starts with island growth of Si and ends with a uniform layer of Si on the substrate. The thickness of the initial transient stage is found to be equal to the thickness of the Pd-silicide layer.

The second stage of growth is characterized by a linear time dependence which is 8 to 10 times slower than the transient stage. The actual rates observed vary with experimental conditions but the activation energy is always the same and has the value of about 4 eV. In cooperation with the University of Illinois, we have established that Auger Electron Spectroscopy (AES) can detect carbon in the Pd₂Si and the amorphous Si layers. The main origin was the carbon crucible in the hearth of the electron gun evaporation system. We established that impurities such as carbon can cause the observed variations in growth rates between different samples.

We further found that, in general, more than two stages of growth can exist. These are defined by equal increments of epitaxial layer growth and decreasing linear rates of growth. The reasons for this discrete behavior were never fully clarified. A probable cause are impurities.

An extension of this work ultimately lead to the recognition that (a) epigrowth is mediated by many silicide layers, and that (b) epitaxial growth should be possible without transport layer, providing that the impurities at the interface between the Si substrate and the amorphous deposited layer, and impurities incorporated in the amorphous layer, are kept sufficiently low. This prediction was later confirmed^(27,29).

We also found that layers of Si formed by SPEG can be doped intentionally⁽⁴⁾. The sample consists initially of an upper layer of amorphous Si (~ 1 μm thick), a very thin intermediate layer of Sb (nominally 5 \AA), and a thin lower layer of Pd (~ 500 \AA), all e-gun deposited on top of a single-crystal substrate (1 - 10 Ωcm , p-type, <100> orientation). After a heating cycle which induces epitaxial growth, electrically active Sb atoms are incorporated into the SPEG layer, as shown by the following facts: (a) the SPEG layer forms a p-n junction against the p-type substrate, (b) Hall effect indicates strong n-type conductance of the layer, and (c) Auger electron spectra reveal the presence of the Sb in the layer.

These results prompted us to investigate the reaction of metal films with polycrystalline Si layers. Solid-phase reactions of metal films deposited on 0.5 μm thick polycrystalline layers of Si were investigated by MeV ^4He BS, glancing angle x-ray diffraction and SEM observations⁽⁵⁾. For the metals Al, Ag, Au, which form simple eutectics, heat treatment at temperatures below the eutectic results in dissolution of the poly-Si layer and growth of Si crystallites in the metal films. Crystallite formation observed at $T \gtrsim 550^\circ\text{C}$ for Ag, $\gtrsim 400^\circ\text{C}$ for Al and $\gtrsim 200^\circ\text{C}$ for Au films. For the metals Pd,

Ni and Cr, heat treatment results in silicide formation. The same initial silicides (Pd_2Si , Ni_2Si and $CrSi_2$), are formed at similar temperatures on single-crystal substrates.

We developed a new technique to trace the relative motion of Si atoms in solid-phase processes. Standard semiconductor-grade Si is irradiated by thermal neutrons in a reactor (we use the Triga reactor at UC-Irvine). The ^{30}Si isotope, which has a natural abundance of 3.1%, is thereby transformed to radioactive ^{31}Si by the $^{30}Si(n,\gamma)$ ^{31}Si reaction. This radioactive nucleus decays to stable ^{31}P by a β^- emission, which can be detected easily with a conventional Geiger-Muller detector. The intensity reached after 1 hr of exposure and the half-life of 2.62 h are nicely matched to the needs of SPEG experiments.

The transport mechanism of Si during solid-phase epitaxial growth (SPEG) in the $Si(xtal)/Pd_2Si/Si(a)$ system was studied using radioactive silicon as tracer. The approach used can briefly be described as follows. Radioactive ^{31}Si is formed by irradiating single-crystal silicon wafers in a nuclear reactor. After depositing Pd and (non-radioactive) Si onto activated single-crystal substrates, Pd_2Si was formed with about equal amounts of radioactive and non-radioactive Si during heating at approximately 400°C. After a second annealing stage (about 500°C) this silicide layer, which moves to the top of the sample during SPEG, is etched off with aqua regia. By measuring the radioactivity in the Pd_2Si etch one can determine whether SPEG has taken place by a dissociation mechanism or not.

From the absence of radioactive ^{31}Si in the etchant solution it was concluded that SPEG takes place by dissociation of the Pd_2Si

layer at the single-crystal interface to provide free Si for epitaxial growth, while new silicide is formed at the interface with the amorphous Si⁽⁶⁾. These results were confirmed by evaporating radioactive silicon onto non-activated silicon substrates before evaporation of Pd and stable amorphous Si and by measuring the activity in the SPEG sample before and after etching off the silicide layer⁽⁸⁾.

This same tracer technique was later applied (by us and others) to other thin-film reactions, such as silicide formation by a single metallic layer (e.g. Ni, Pd), or bilayers (e.g. Cr on Pd on <Si>), to the determination of self-diffusion coefficients for Si in silicides, and the moving specie in the thermal oxidation of Si and silciides.

A study of the growth of epitaxial layers of Si on Si substrates by the process of SPEG through Pd₂Si on <111>-oriented substrates brought to light an interesting variation of the growth process which leads to a heteroepitaxial growth of Si and Pd₂Si⁽⁷⁾. The effect results from the fact that the structure of Pd₂Si depends on the orientation of the Si substrate. On <100> substrates, the Pd₂Si is polycrystalline, with a c-axis of the grains preferentially oriented parallel to the <100> axis of the substrate. On <111>-oriented substrates, the Pd₂Si layer grows epitaxially and forms a single-crystal layer with the substrate; the c-axis is again co-linear with the <111> axis of the substrate. If a thin layer of Pd is deposited on a <111>-oriented substrate and covered by a layer of amorphous Si that is done for SPEG samples, the first annealing stage

at about 400°C for 30 min converts the Pd to Pd_2Si by reaction with the Si at both interfaces. That part of the silicide layer formed by reaction with the <111>-oriented substrate will be single crystalline, while the other part formed with the amorphous Si layer is not. The Pd_2Si layer is thus divided into two sublayers: a single crystalline part, and a polycrystalline part. On the second annealing stage at about 500°C, one observed that only the polycrystalline layer dissociates to promote SPEG. The epitaxial growth of Si then takes place on the single-crystal Pd_2Si , and a heteroepitaxial Pd_2Si -Si structure results.

The process of heteroepitaxial growth of silicon on silicides has recently attracted renewed interest, particularly with $NiSi_2$ and $CoSi_2$, because these silicides are cubic and match the crystallographic cell of Si very closely.

The redistribution of implanted As and Sb following metal-silicide formation of Pt, Pd and Ni has been studied. The phases of the silicides used were $PtSi$, Pd_2Si and $NiSi$. Investigations with BS analysis showed that after the formation of the silicides the Sb was always found in the silicide layer near the surface of the samples whereas $PtSi$ and Pd_2Si caused a partial rejection of As for implanted doses of $2 \times 10^{15} cm^{-2}$ and higher. No rejection of As was found after the formation of $NiSi$. The results are discussed in terms of solid solubilities and impurity-metal compound formation⁽¹¹⁾. These results have important implications in the fabrication of ohmic contacts and the adjustments of the heights of Schottky barriers on silicon that still await clarification.

In a later study, we also investigated the effect of oxygen impurities on the growth kinetics of nickel silicide⁽²²⁾. We found that oxygen in the Ni film forms SiO_2 when it comes into contact during annealing with the Ni_2Si front of the growing silicide layer. The identification of the chemical state of the silicon at that interface was done by ESCA. When the total accumulated amount of SiO_2 reaches the equivalent of a layer of about $20-30 \text{ \AA}$, the Ni_2Si reaction comes to a premature halt. This effect, too, is potentially of practical significance, as we showed later that the contact resistivity of such a system is fairly low.

Another study combined SPEG with ion mixing⁽¹⁶⁾. Epitaxial regrowth of an amorphized Si layer on a <100> Si crystal held at 200-400°C was achieved under bombardment with Si, Kr, or Xe ions. Channeling measurements with MeV He ions show the regrowth proceeds from the amorphous-crystalline interface, and has an initially linear dose dependence. The annealing beam, however, introduces additional damage centered at or beyond the ion range. Amorphous layers obtained by low temperature self-ion bombardment regrow much more readily than amorphous deposited layers.

We also proposed a rule for the sequence of the phases formed by thin metal films on silicon upon thermal annealing⁽³⁰⁾. After the formation of the first silicide phase, which generally follows the rule proposed by Walser and Bené, the next phase formed at the interface between the first phase and the remaining element (Si or metal) is the nearest congruently melting compound richer in the unreacted element. If the compounds between the first phase and the remaining element are all noncongruently melting compounds (such

as peritectic or peritectoid phases), the next phase formed is that with the smallest temperature difference between the liquidus curve and the peritectic (or peritectoid) point. The rule correlates well with observations.

2. 400 kV Linear Accelerator System.

A 400 kV Accelerators Incorporated implantation system was acquired under this contract, installed, tested and accepted on May 27, 1977. Improvements were made continuously: we installed closed-loop water cooling to eliminate corrosion problems in the magnet cooling coils; redesigned the thermal protection circuit of the magnet power supply and the diffusion pumps; earthquake proofed the whole system (a substantial undertaking); built a target end station for cooling and heating of the target during implantation; designed and installed a reliable current integration system^(13,14), and in parallel to it, developed an absolutely calibrated Au target for BS measurements^(12,13), calibrated the energy by a (p, γ) reaction; installed a TV monitor for the source settings and installed a second backscattering spectrometry leg. This system was the basis of most of the work performed in the latter parts of the contract.

3. High-Dose Implantations and Ion Mixing.

An early study investigated the influence of noble gas atoms on the epitaxial growth of implanted and sputtered amorphous silicon⁽¹⁷⁾. We compared the epitaxial regrowth of amorphous silicon which had been implanted with Ne, Ar or Kr at annealing temperatures below 600°C. The aim was to determine if silicon sputtered on single-crystal silicon with an appropriate sputtering gas could grow epitaxially. We showed that this process is not possible because of the high concentration of gas included in sputtered films. We evaluated the maximum concentration of Ne, Ar and Kr in silicon, above which the amorphous layer does not regrow epitaxially.

A similar study investigating the high-dose implantation of noble gases in a thin metallic film (Cr, V, Ni, Ti or Pd) on a silicon substrate led to the recognition of bubble formation within the sample⁽¹⁵⁾. Rutherford backscattering measurements and cross-sectional transmission electron microscopic analyses indicated that the gas bubbles were formed predominantly near the interface between the Si substrate and the surface layer. In addition, pronounced mixing between Si and metal was observed. Both bubble formation and Si-metal intermixing were interpreted in terms of ion-induced atomic migration within the collision cascade around the ion track. With increasing ion dose, the number of gas atoms trapped in the bubbles at the interface continued to grow and the surface layer peeled off at high doses.

Ion mixing investigations continued with a study of the formation of palladium silicide phases by implantation of Si ions into evaporated palladium films on two different substrates (Si and SiO_2). The amount and phase of silicide formed depend on the implantation temperature, substrate type and the penetration of the ions relative to the substrate depth. Provided the ions do not penetrate to the substrate, it is found that Pd_2Si was formed for both types of substrate, even at 150°K. When the ions just reach the Pd/substrate interface, the sample temperature and substrate type become important. For the SiO_2 substrate, the amorphous alloy Pd_4Si forms at low temperatures. With the Si substrate, Pd_2Si forms under all conditions and there is a large incorporation of silicon from the substrate into the palladium, to an extent dependent on the temperature. This effect was explained in terms of radiation enhanced diffusion, possibly assisted by dynamic cascade mixing. In the SiO_2 substrate case, oxygen was

thought to play an important role in determining the formation of phases.

To get insight into the ion mixing process, marker experiments were performed. Samples consisting of a thin Pt (or Si) marker a few tens of angstroms thick buried at different depths in a deposited Si (or Pt) layer were bombarded with Xe and Ar ions. Significant spreading of the marker was observed as a result of ion irradiation. The amount of spreading depended upon the ion species and the depth of the marker. The amount of spreading was in good agreement with the amount of energy deposited in elastic displacements⁽²⁴⁾

The magnitude of the irradiation-induced-mixing of thin buried layers depends on the metallurgy of the marker system. To show this, complementary structures were made with Ge, Ni or Pd layers as markers in Si and Si layers as markers in Ge, Ni or Pd⁽²¹⁾. For the Si-Ge system the linear cascade model gives a good description of the spreading in either configuration. A thermally dependent mixing is observed in the case of Si in metals and Pd in Si at room temperature. This is attributed to radiation-enhanced diffusion mechanisms. At low temperatures, a thermally independent collision cascade mixing is observed in all cases.

In the temperature-independent regime, it was found that the diffusion length squared, Dt , increases linearly with ion dose ϕ for Kr⁺ ranging from 6.3×10^{14} to 2.0×10^{16} Kr/cm² for thin Ge, Sb and Pt marker in amorphous Si. This suggests that Dt/ϕ is a pertinent parameter to characterize the interaction of ion beams with amorphous Si^(31,33). Dt/ϕ was found to be independent of the temperature during irradiation from ~ 80K to ~ 300K for Ni, Ge, Sn, Sb, Pt and Au

markers in amorphous Si; however, mixing of Pd is strongly temperature-dependent over the entire range investigated.

An analytical model was developed that faithfully describes and quantitatively predicts the magnitude of the observed effects on the basis of the theory of random flights and elementary range theory⁽³²⁾. The model predicts that the mixing is analogous to thermal diffusion with an effective diffusion coefficient that is proportional to the ion flux and the linear energy deposition density F_D . The diffusion coefficient depends on the marker and matrix materials through their atomic masses, atomic numbers and the effective displacement energy of the marker atoms. The model was derived for dilute impurities in a host matrix, but an approximate method for extension to higher concentrations was suggested. The model was shown to be applicable in situations in which the energy deposited per atom is not a large fraction of the heat of fusion of the solid.

4. Very Thin Si Films and Related Investigations.

A procedure was developed to prepare large-area self-supporting silicon thin crystals with (100), (110), and (111) orientations⁽²⁶⁾. Windows up to 2 cm in diameter and 1400 to 20,000 Å in thickness were produced. From backscattering measurements on a 1700 Å thick samples, it was found that the uniformity of the sample varies only by ± 60 Å across the window area. The samples are durable; the thin crystals can be made amorphous by ion implantation up to 1/3 of the original thickness or can be heated to 1000°C. Layers of SiO_2 and metal silicides can also be grown on these thin crystals by thermal annealing up to 850°C.

These samples were used in a study of the Si-SiO₂ interface by MeV ion-channeling in collaboration with the Bell Laboratories group led by L. C. Feldman⁽²³⁾. At Caltech, the thin crystals were used to study the Ni/Si and NiSi₂/Si interfaces.

In the Ni-Si interface, channeling measurements were used to locate preferential sites of monolayers of Ni atoms deposited onto Si crystalline substrates⁽²⁸⁾. The Ni scattering yield showed dips or peaks in the angular scan measurements when channeled along the major crystallographic axes and planes of Si. The results showed that 2×10^{15} Ni/cm² occupied Si tetrahedral sites at the intermixed Ni-Si interface. In another investigation, epitaxial NiSi₂ layers were grown on 5000 Å silicon single-crystals at 850°C. The epitaxial quality of the silicide and the silicon-silicide interface were characterized by MeV ⁴He channeling with the beam incident on either side of the sample⁽²⁵⁾. The mosaic structure (standard deviation $\sim 0.2^\circ$) of the silicide was characterized by measurements with NiSi₂ on the front side (facing the incident beam). Measurements with the NiSi₂ on the back side were used to show that the atomic disorder at the NiSi₂/Si interface is less than 1×10^{16} Si atoms/cm². Transmission experiments were also used to determine small misorientations of the epitaxial layer with respect to the Si substrate.

The possibility to nondestructively investigate buried interfaces of silicon single-crystals by this technique is a major step forward. These very thin silicon crystal layers are also useful in deciphering complex BS spectra because two independent measurements can be performed from the front and the back side of the sample. Their application in ion mixing experiments and channeling studies is similarly successful, as ongoing experiments demonstrate.

III. Scientific Collaborators

The following were engaged in research under project
N00014-75-C-0912 at the California Institute of Technology:

<u>Investigator</u>	<u>Status</u>	<u>Present Address</u>
J. S. Best	Student	IBM, San Jose, California
R. J. Blattner	Collaborator	CHARLES EVANS & ASSOCIATES San Mateo, California
R. L. Boatright	Student	U.S. Air Force Academy, Colorado Springs, Colorado
C. Canali	Research Fellow and Collaborator	Istituto Di Fisica, Modena, Italy
G. E. Chapman	Research Fellow	Riverina College of Advanced Education, Wagga Wagga, Australia
N. W. Cheung	Student and Research Fellow	University of California, Berkeley, California
C. A. Evans, Jr.	Collaborator	CHARLES EVANS & ASSOCIATES San Mateo, California
L. C. Feldman	Collaborator	Bell Laboratories, Murray Hill, New Jersey
R. Fernandez	Technician	Caltech
I. Golecki	Research Fellow and Visiting Assoc.	Rockwell International, Anaheim, California
R. Gorris	Technician	Caltech
M. G. Grimaldi	Research Fellow	Istituto Di Struttura di Materia, Catania, Italy
P. J. Grunthaner	Student	Jet Propulsion Laboratory, Pasadena, California
J. M. Harris	Student	Sandia Laboratories, Albuquerque, New Mexico
S. H. Harrison	Collaborator	Sandia Laboratories, Albuquerque, New Mexico

Scientific Collaborators (Continued)

L. S. Hung	Research Fellow	Cornell University, Ithaca, New York
S. S. Lau	Senior Research Fellow	University of California, La Jolla, California
Z. L. Liau	Student	Lincoln Laboratory, M.I.T., Lexington, Massachusetts
R. M. Lindstrom	Collaborator	Sandia Laboratories, Albuquerque, New Mexico
M. Mäenpää	Research Fellow	Caltech
J. J. Mallory	Technician	-
S. Matteson	Research Fellow	Texas Instruments, Dallas, Texas
J. W. Mayer	Professor	Cornell University, Ithaca, New York
J. O. McCaldin	Professor	Caltech
G. Mezey	Research Fellow	Central Research Institute for Physics, Budapest, Hungary
K. Nakamura	Research Fellow	Nippon Electric Co., Ltd., Kawasaki, Japan
R. G. Newcombe	Collaborator	McMasters University, Hamilton, Ontario, Canada
M-A. Nicolet	Professor	Caltech
C. Norris	Secretary	-
J. O. Olowolafe	Student and Collaborator	University of Ife, Ile-Ife, Nigeria
B. M. Paine	Research Fellow	Caltech
M. Parks	Secretary	Caltech
R. Pretorius	Research Fellow	Southern Universities Nuclear Institute, Faure, South Africa

Scientific Collaborators (Continued)

J. Roth	Research Fellow	Max-Planck-Institut für Plasmaphysik, Garching bei München, West Germany
D. M. Scott	Student	Caltech
T. E. Seidel	Collaborator	Bell Laboratories, Murray Hill, New Jersey
R. Shima	Collaborator	Jet Propulsion Laboratory, Pasadena, California
P. J. Silverman	Collaborator	Bell Laboratories, Murray Hill, New Jersey
I. Stensgaard	Collaborator	Bell Laboratories, Murray Hill, New Jersey
D. A. Thompson	Visiting Associate and Collaborator	McMasters University, Hamilton, Ontario, Canada
D. G. Tonn	Technician	Caltech
B. Y. Tsaur	Student	Lincoln Laboratory, M.I.T., Lexington, Massachusetts
W. F. Tseng	Research Fellow	Naval Research Laboratory, Washington, D. C.
K. N. Tu	Collaborator	IBM, Yorktown Heights, New York
M. Wittmer	Research Fellow	IBM, Yorktown Heights, New York

IV. Scientific Writings

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